

EPA Method 8270 Analyzer

Purpose: To provide a list of simple steps to successfully install the EPA Method 8270 Productivity Solution to transform the TRACE GC Ultra, AS 3000 autosampler, and DSQ II into an EPA Method 8270 analyzer.

Methods: The AS 3000 autosampler, TRACE GC Ultra, and DSQ II are set up for a split injection with EI Full Scan acquisition. Electronic copies of the Xcalibur instrument control and processing methods (*Emethods*) are directly linked to these files:

- EnviroLab Forms Master Method: *EPAMethod8270.mext*

- Daily batch templates: *EPAMethod8270Curve.utm*, *EPAMethod8270MDL.utm*, *EPAMethod8270MVD.utm*, and *EPAMethod8270Daily.utm*, which are configured for automated report generation.

Results: Four batches to run:

1. Calibration Curve: to establish the linear working range for the method.
2. MDLS: to establish the precision and accuracy of the system.
3. MVD: to establish precision of mid level replicate injections of all target compounds (optional).
4. Daily batch: to check the % drift of the response factors (RF) of a mid point standard against the mean RF from the initial calibration curve.

Introduction

The optimum parameters for this analyzer: injection, separation and detection have all been previously determined for you to facilitate the start up of your new instrument in your lab.

Materials & Methods

This guide provides three Quick Start steps for this kit:

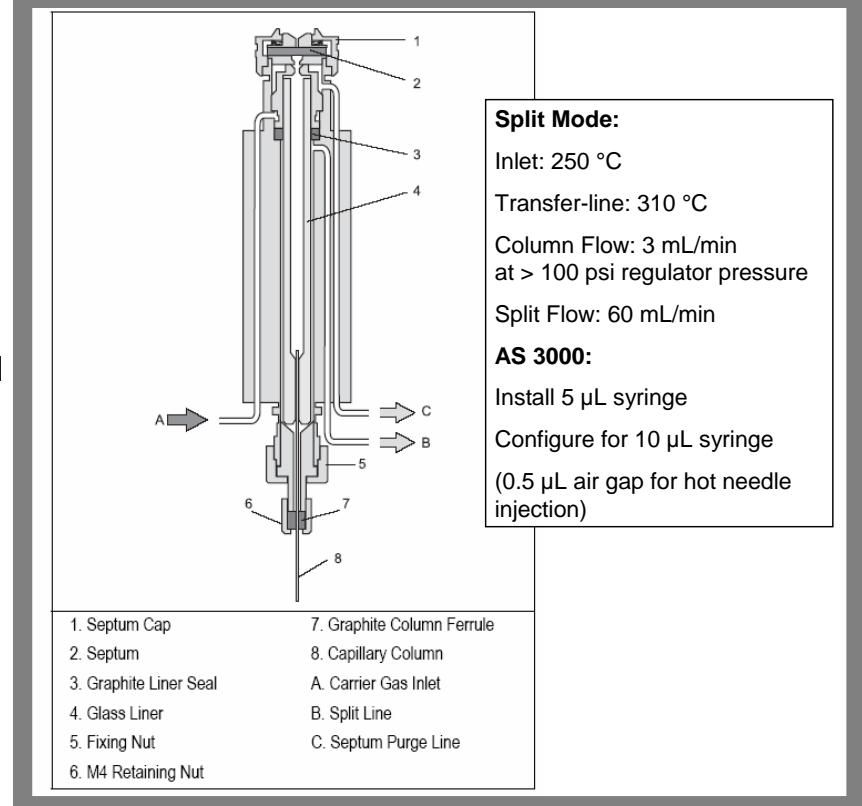
- [Step 1 Installing the Kit](#)
- [Step 2 Tuning the DSQ II for EPA Method 8270 \(DFTPP\)](#)
- [Step 3 Starting the Analysis](#)

Look for the *Emethods* supplied on the CD and validation data for comparison to the data that you will generate on your new analyzer. FAQs are included in the SOP, PN 120291-0001.

Step 1 Installing the Productivity Solution

1. Put the 5 μ L syringe in the AS 3000.
2. Cool the inlet and transferline.
3. Install the 5 mm Splitless liner and BTO septum.
4. Place a no hole ferrule into the transferline of the mass spec.
5. Restart the mass spec with the transferline temperature set to 150 $^{\circ}$ C.
6. Insert the capillary column to 40 mm (Figure 1).
7. Set the carrier gas to constant flow at 3 mL/min and the vacuum compensation to OFF.
8. Run the column evaluation ($k = 1.7 \pm 0.2$).
9. Pre-condition the capillary column as specified by the manufacturer.
10. After conditioning the column, vent the mass spec.
11. Install the column to 2-3 mm beyond the end of the transferline tip.
12. Turn the mass spec on and set the vacuum compensation to ON.
13. After pumping for one hour, run the column evaluation ($k = 1.7 \pm 0.2$).
14. Leak test the system.

FIGURE 1. Column Installation at 40 mm



Step 2 Tuning for EPA Method 8270

Running Autotune and Target Tune

Open the Tune Window:

1. Set the source temperature to 275 °C.
2. Install the Closed Exit Ion Volume (CEI).
3. Set the EI Source ion volume to CEI.
4. Run Autotune at 25 µA.
5. Compare the Autotune Report to the one in Figure 2.
6. Run Target Tune at 25 µA
7. Compare the Target Tune Report to the one in Figure 3.
8. Save the Target Tune Report to a new name, for example, *DFTPP.dsqtune*.
9. Run the Xcalibur sequence: *StartUp.sld*
10. Make two injections of DFTPP at 50 ng/µL and four injections highest level standard (160 ng/µL).
11. Review the Tune report from an imported spectrum in EnviroLab Forms.
12. If any criteria fails, go to the heading labeled *If DFTPP Fails*.

FIGURE 2. Autotune Report

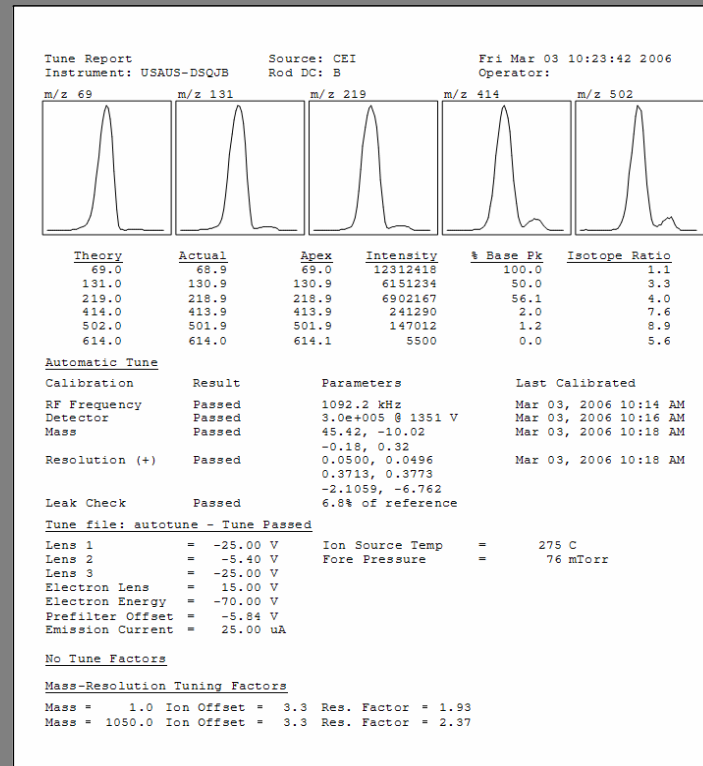


FIGURE 3. Target Tune Report

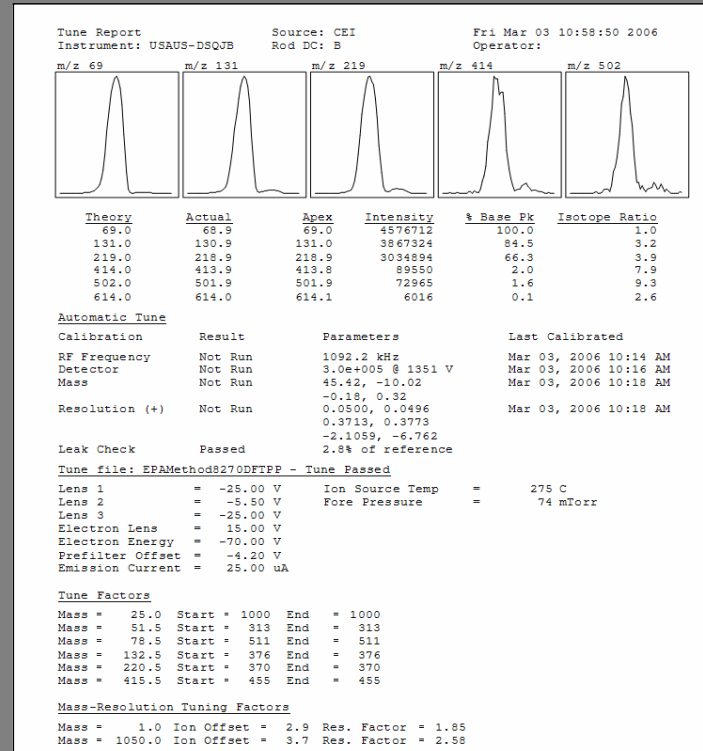


FIGURE 4. DFTPP performance solution 50 ng/µL

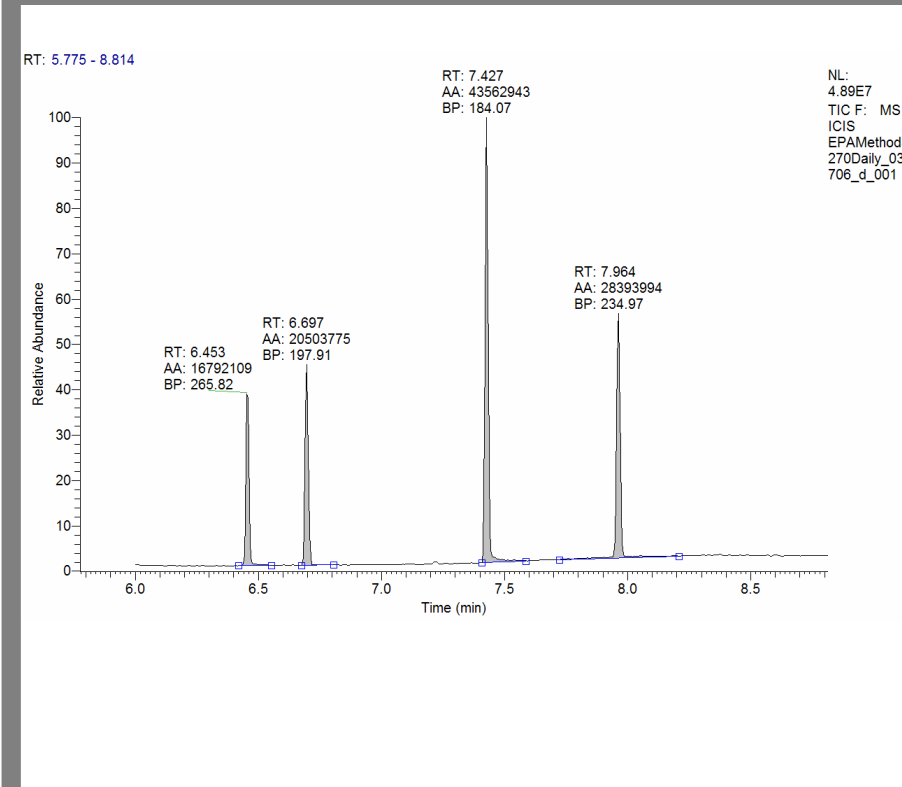
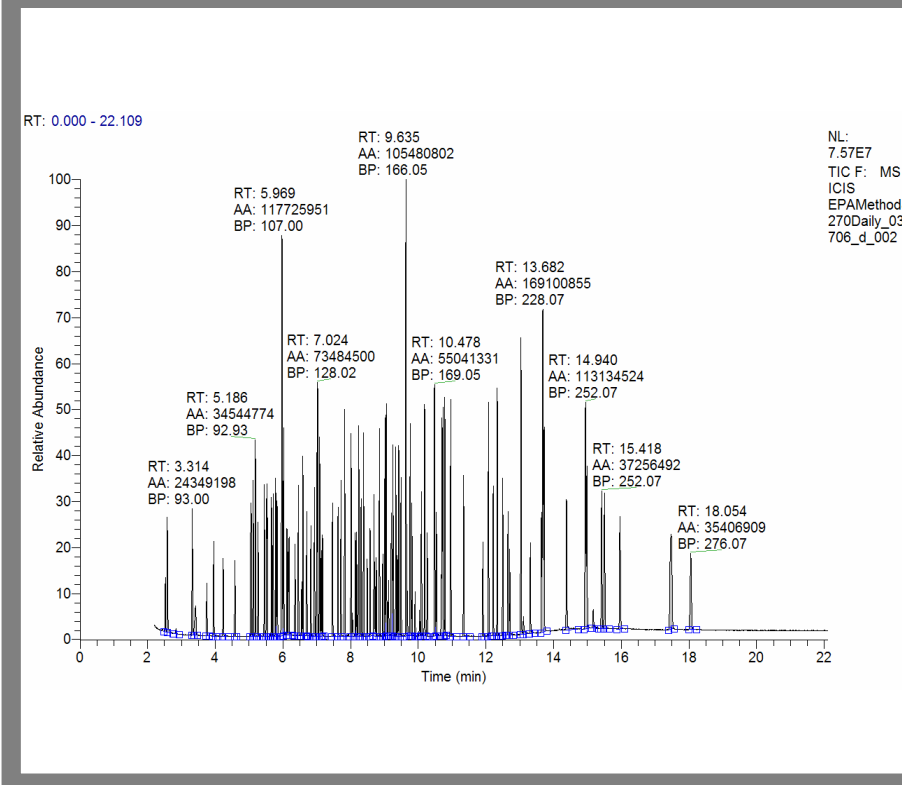


FIGURE 5. Mid-level standard 40 ng/µL



Step 2 Tuning for EPA Method 8270 (continued)

13. Typical TICs for the tuning solution (DFTPP) and mid-level standard are shown in Figures 4 and 5.

If DFTPP Fails:

The Target Tune file tunes the mass spectrometer to meet DFTPP tuning criteria. If you fail any of the ions listed in Table 1, refer to the **Action Items** and re-inject DFTPP until it passes. Usually only one or two ions will fail the criteria. Change one parameter at a time in the Target Tune File (refer to Figure 6) and re-inject until it passes.

TABLE 1. Corrective Action for Meeting DFTPP

Mass Range	DFTPP ion (m/z)	Cal gas Ion (Tune Targets listed in Figure 6)	Default	Action
51.5-78.5		69	Fixed 100%	
25-51.5	51	50	1.1	1
78.5-132.5	127	131	90	2
132.5-220.5	198	219	70	3
220.5-415.5	275	414	2.0	4
415.5-1050	442	502	1.3	5
		Tune Limits		
		Maximum Peak Width	0.95	
		Measure Peak Width	10%	
	68, 70 (low mass resolution)			6
	365 (threshold)			7
	197, 199 (mid mass resolution)			6
	441, 443 (high mass resolution)			6

Action Items:

1. If 51 m/z (listed in Table 1) is too low, raise the default value by 0.2 (see Figure 6.) If it is too high, lower it by 0.2.
2. If 127 m/z (listed in Table 1) is too low, raise the default value by 20, and lower by 20 if too high.
3. If 198 m/z (listed in Table 1) is too low raise the default value by 20, and lower by 20 if too high.
4. If 275 m/z (listed in Table 1) is too low, raise by 1.0; and lower it by 1 if it is too high.
5. If 442 m/z (listed in Table 1) is too low, raise it by 1.0, if it is too low high lower it by 1.0.
6. For all failures of mass resolution, adjust the maximum peak width value. This adjustment should be by raising it by 0.05. For more adjustment, you may change where the peak width is measured at 10% of the height of the peak or lower.
7. If 365 m/z is too low, turn up the detector gain by + 0.2 (0.8- 2).

3. Starting the Analysis

After meeting the tuning criteria for DFTPP, you are ready to run the following four batches:

- Day 1: *EPAMethod 8270Curve.utm* (6 level calibration curve 5-160 ng/μL).
- *EPAMethod8270MVD.utm* (optional replicate injections of mid-level standard (40 ng/μL).
- Day 2: *EPAMethod8270MDL.utm* (replicate injections of 1 ng/μL).
- Day 3: *EPAMethod8270Daily.utm* (QC set of Tune, Check standard, Blank, LCSCSD, MSMSD, TIC library search of unknown).

Calibration Curve

1. In EnviroLab Forms open the Master Method:
EPAMethod8270.mext.
2. Create a new batch by opening the Curve Batch Template:
EPAMethod8270Curve.utm.
3. Export the new batch to Xcalibur.
4. Load the AS 3000 autosampler sample tray:
 - 60181-205: Tuning Standard
 - Blank (Take 1 mL of methylene chloride and spike in 10 μL of 60181-206 (4000 ng/μL internal standards)
 - One each of the curve (C1-C6: 60181-202)
5. Initiate the acquisition.

MDLs

1. Create a new batch by opening the MDL Batch Template:
EPAMethod8270MDL.utm.
2. Export the new batch to Xcalibur.
3. Load the AS 3000 sample tray:
 - 60181-205: Tuning Standard
 - Blank
 - 60181-203: Mid-level check standard (40 ng/μL)
 - 60181-204: 10 replicate vials of MDL standard (1 ng/μL). Generally designed as IDL (Instrument Detection Limit). For true MDL, spike these standards into matrix and extract.
4. Initiate the acquisition.

Daily

1. Create a new batch by opening the Daily Batch Template:
EPAMethod8270Daily.utm.
2. Export the new batch to Xcalibur.
3. Load the AS 3000 sample tray:
 - 60181-205: Tuning Standard
 - Blank
 - 60181-203: Mid-level check standard (40 ng/μL)
 - Prepare LCS, LCSD, Sample (Spl) and Spl-MS, Spl-MSD, and Spl for library search
4. Initiate the acquisition.

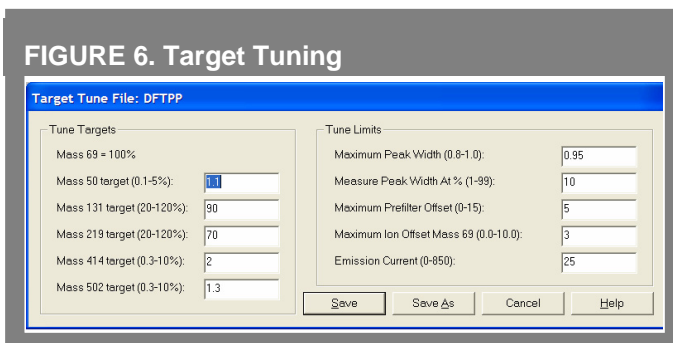


FIGURE 6. Target Tuning

Reorder Guide:

Table 2 lists the consumables for the kit which can be ordered by calling: 1-800-532-4752 or accessing our website: <http://www.thermo.com>. Call Technical Support at 1-800-685-9535.

TABLE 2. Consumables

EPA 8270 Productivity Solution Re-order Guide		
Item	Part Number	Description
EPA Method 8270 Kit	120291-Kit	
SOP Manual	120291-0001	Standard Operating Procedures
How To Manual Quick Start Guide	120291-0002 120291-0003	How To manual 4-page, 11x17, laminated Quick Start Guide
Closed Exit Ion Volume	119650-0221-T	
Closed Exit Ion Volume Holder	70001-20532-T	
Column Ferrules for Inlet	29053488	(1pk of 10)
Column, Split primary	26RF223P	0.25 mm x 30 meter, 0.5 µm
Column, Splitless ¹	26RF296P	0.25 mm x 30 meter, 1.0 µm
Inlet Septa	31303211	BTO (50 ea.)
Liner Seal graphite	29033406	(1 pk of 10.)
Splitless Liner	45350033	5 mm (1 pk of 5.)
Transferline ferrule for column	29033496	(1 pk of 10.)
Transferline no hole ferrule	290VT155	(1 pk of 10.)
Vial	60180-565	Amber with Teflon red rubber septa, 1 pk of 100
Syringe	36500505	2, autosampler syringe, 5 µL
Semivolatile Method Kit ²	60181-201	22 ampules
Internal std.	60181-206	1 x 1 mL (4000 µg/mL)
MDL std.	60181-204	5 x 1 mL (1 µg/mL)
Semivolatile Calibration Curve	60181-202	6 ampules per kit (C1-C6) 1 mL each (5,10,20,40,80,160 µg/mL)
Tuning std.	60181-205	5 x 1 mL (50 µg/mL)
Working daily std.	60181-203	(C4) 5 x 1 mL (40 µg/mL)

1. Not part of Kit
2. Productivity Solution includes MSDA Documents